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Contributed Review: A review of the investigation of rare-earth dopant profiles in optical fibers

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Rare-earth doped optical fibers have captivated the interest of many researchers around the world across the past three decades. The growth of this research field has been stimulated primarily through their application in optical communications as fiber lasers and amplifiers, although rare-earth doped optical fiber based devices are now finding important uses in many other scientific and industrial areas (for example, medicine, sensing, the military, and material processing). Such wide commercial interest has provided a strong incentive for innovative fiber designs, alternative glass compositions, and novel fabrication processes. A prerequisite for the ongoing progress of this research field is developing the capacity to provide high resolution information about the rare-earth dopant distribution profiles within the optical fibers. This paper constitutes a comprehensive review of the imaging techniques that have been utilized in the analysis of the distribution of the rare-earth ion erbium within the core of optical fibers. *Published by AIP Publishing.* [http://dx.doi.org/10.1063/1.4947066]

I. INTRODUCTION

Rare-earth (RE) doped optical fibers were first reported in the 1960s, where the goal was to exploit the optical activity of the RE elements to create a laser or optical amplifier.[1–3] From that time until the late 1980s, such fibers were primarily a research platform, which did not mature into commercial products. This changed very dramatically with the development of the erbium (Er)-doped fiber amplifier (EDFA),[4] which provided the first realistic solution to the rising critical need for an all-optical amplifier within optical communication systems. The success of the EDFA and the subsequent advancement of high power fiber lasers and amplifiers for telecommunications, industrial materials processing (welding, drilling, cutting, micromachining), medical (imaging, surgery, therapeutics), military (weapons, imaging, ranging), and environmental monitoring (fiber optic sensing) applications, spawned an industry that even today maintains continuous growth. Innovation in this arena continues, including the exploration of novel fiber fabrication techniques,[5] the search for alternative dopants and hosts,[6] and the development of improved fiber designs and devices.[7] However, there currently exist significant roadblocks to some areas of innovation due to limitations in characterizing the RE distribution within the fiber core. By way of illustration in a recent review of silica optical fibers doped with nanoparticles for fiber lasers and broadband sources,[8] it was concluded that "the next progress of nano-characterization methods will represent a valuable contribution to this field. Modification of available nanoparticle study techniques for investigation of small cores of active fibers and development of novel analytical instruments represent a specific task." Through the present review, it is expected that RE fiber fabricators and users will acquire a ready guide concerning available characterization techniques and in addition it is hoped that this review will serve as a launching point from which new and improved techniques will emerge.

Optimizing the properties of RE-doped optical fibers and devices has become increasingly important; critical to this quest is the ability to thoroughly characterize a number of post-fabrication physical parameters that are intrinsically related to the fiber device performance. The most important parameters are the fiber glass material, the waveguide characteristics, and the RE dopant concentration profile. An accurate knowledge of the latter has been found to be vital for the optimal design and operation of RE doped fiber lasers and amplifiers.[9–16] This has motivated the exploration of various techniques in order to provide specific information relating to the confinement of the RE dopants in the fiber core. These techniques vary; some may be applied to the preforms from which the fiber is drawn, whilst others have the capacity to interrogate the fiber itself. This review summarizes the various means of characterizing the RE distribution in single or few mode optical fiber, with the major focus being on erbium doped fiber. It is acknowledged that the dimensions of fiber used for many high power fiber lasers are large and therefore the critique given in this review may not universally apply; however, the techniques discussed here will still be of benefit for researchers in that field. As a general rule, this review has relied on the published literature of measurements conducted on erbium doped preforms and optical fiber. Therefore, the reported resolution of the various techniques comes from this body of literature and it is acknowledged that superior resolution may indeed be possible for any particular technique through a variety of factors, which include: the parameters governing the focus of the instrument, efforts to reduce detector noise, and the erbium concentration being measured. Notwithstanding this limitation, it is hoped that this review will prove both valuable and timely.

In Section II, the physical principles and performance of several of the better known techniques that have been used for the acquisition of the concentration mapping of RE dopants in fiber preforms are reviewed. Section III provides a similar overview, however, specifically dedicated to

techniques applied directly to the much smaller doped core of a drawn fiber. Special emphasis has been given to optical microscopy based techniques used to determine the RE dopants in fibers due to their cost effective operation and attractiveness for further development.

II. RE DOPANT DETERMINATION IN OPTICAL FIBER PREFORMS

During the production of optical fibers, the concentration of RE dopants (and other network modifiers) and their distribution, together with the refractive index (RI) profile and other parameters, are most commonly measured prior to the drawing process. The resultant dopant concentration and distribution is then scaled down to the dimensions of the drawn fiber assuming that there are no defects or redistribution of the constituent elements affecting the resultant fiber profile. To date, some of the most important techniques employed to reveal the location of dopants in bulk samples such as fiber preforms are based on the use of methods that are commonly applied in the field of analytical chemistry and material science. These techniques include; secondary ion mass spectroscopy (SIMS),[9,17,22] inductively coupled plasma mass spectroscopy or inductively coupled plasma atomic emission spectroscopy,[17,18] and X-ray microprobe analysis.[11] In addition, the confinement of dopants in fiber preforms has been examined using the electron probe microanalysis (EPMA) method and its counterpart systems.[19,20] In other cases, optical means such as fluorescence-induced laser scanning microscopy (LSM) or micro-fluorescence profiling have been utilized.[21]

It is import to note that in most of the published literature, there is at best modest detail regarding the particulars of the profiling measurements made such as the relevant physical parameters and/or the conditions under which the investigation of the dopant distributions was made (see, for example, Refs. 9 and 17). This comment is not offered as a criticism rather it acknowledges that many investigators have been content with a knowledge of the relative dopant distribution so have not made efforts toward the direct determination of the RE dopant concentration profile. It is not surprising therefore that there is limited detail regarding parameters such as image resolution, sample preparation, accuracy of the measurements made, complexity of the techniques used, or the cost of the imaging system. However, as RE fiber systems mature, the need for superior characterization of their various material parameters and properties has increased along with the desirability of knowing the precision and accuracy of the information produced.

A. Secondary ion mass spectroscopy

SIMS involves the bombardment of the surface of a solid sample placed in a vacuum chamber with high energy ions (primary ions). This causes ions from the surface of the sample (secondary ions) to eject or sputter. The ejected ions are collected using a mass spectrometer and their mass to charge ratios are analyzed to allow identification of the ions in the sample. The detected number of backscattered ions gives a relative measure of the concentration of that ion in the sample, while by scanning the focused ion beam across the whole sample surface, it is possible to obtain the lateral distribution of the elements. The spatial resolution of such a scan depends on the primary ion beam diameter; resolutions of the order of 1–2 μm are possible.[22]

SIMS was used to measure the Er, aluminum (Al), and phosphorous (P) distributions across (perpendicular) and along (parallel) the axis of Er doped silica based preforms that had been fabricated with the modified chemical vapor deposition (MCVD) and solution doping techniques.[22] Preform slices were first cut at different sections along the length of the preforms and then polished prior to imaging. Line scans were carried out using a focused primary ion beam (~10 μm beam diameter) produced by a duoplasmatron, in conjunction with, a Wien filter operated with a 50 nA current of O_2^+ at 4 keV. Among the many outcomes, it was found that the Er and Al dopant distributions vary independent of each other across and along the fiber preform with Er exhibiting the greatest variation. The Er concentration was found to vary along a 4 cm section of the preform by as much as a factor of two. Therefore, one would logically expect a similar variation in the Er concentration over a 200 m length of fiber, which would typically be drawn from a 4 cm section of the preform. It is worth noting that optical fiber fabrication methods have improved significantly since this time (1989) and that significant variation of constituent concentrations along the fiber length is now highly unlikely. The P dopant profile exhibited a dip in the center of the core, indicative of the volatile nature of the phosphorous oxide during the preform collapse stage.

Valuable information was collected for the bulk sample under examination; however, the limited lateral spatial resolution of the system means that this technique may only be applied to the investigation of fiber preforms and bulk samples. The resolution is insufficient for analysis of drawn fiber.

B. X-ray microprobe spectroscopy (XMS)

XMS involves using a finely focused X-ray microbeam to illuminate the sample under examination. X-ray beams can be used to analyze data in one of two ways; diffraction or fluorescence analysis. Diffraction analysis uses the detection of scattered X-rays to give information about the structure and crystallography of the sample. X-ray diffraction is similar to other diffraction techniques such as electron or neutron diffraction with a few notable advantages, one of which being that the X-ray wavelength is similar to the atomic spacing. This method should allow for detection of clusters within the RE-doped samples by measurement of X-ray absorption fine structures that provide localized structural information about the element of interest.[23]

Optical fiber preforms with their germanosilicate core regions doped with small quantities of Er ions have been studied using Extended X-ray Absorption Fine Structure Spectroscopy (EXAFS). A circular X-ray beam of 100 μm diameter was used to gather information as a radial function of position across the core region of the preform. The samples studied were manufactured using the MCVD and solution doping processes and consisted of transverse slices of fiber preform

that had been ground and polished down to a thickness of about 100 μm.

Absorption maps of the Er and germanium (Ge) distribution across the core region of the preforms were obtained showing a correlation between the distributions of the two atom types, i.e., the Er ions introduced via the solution doping technique followed the Ge concentration profile established during the core soot deposition phase of the MCVD process. The results show that evaporative losses affecting the Ge dopant also affected the Er incorporated into the preform core. Therefore, it was stated that if the assumption that the Er tracks the Ge profile is valid, measurements of the RI profile, which are dependent upon the Ge content of the glass, would also be representative of the Er concentration.

Fluorescence analysis relies on using an X-ray to excite the sample causing emission of a characteristic fluorescence that is used to identify elements within the sample. XMS is inherently non-destructive and can provide information with a spatial resolution of the order of the size of the circular X-ray beam used to analyze the sample (typically 100 μm).[23] Similar to the SIMS technique, EXAFS is suitable for preform analysis but not drawn fiber. In addition, since high intensity X-rays can be produced only by synchrotrons, this technique is not fiscally practicable for the routine investigation of optical fibers. XMS instruments also require special X-ray focusing optics adding to the complexity and cost.

C. Electron probe microanalysis

A similar technique to X-ray fluorescence analysis is EPMA. This technique is commonly referred as energy dispersive X-ray analysis (EDAX) or wavelength dispersive analysis of X-rays (WDX). The operation of this analytical system is based on the fact that the resulting X-rays emitted from a sample after being bombarded by a finely focused electron beam have energies and wavelengths characteristic of the elements within the sample. Therefore, from the analysis of the resulting X-rays, the sample's elemental composition can be identified.

In addition, the EDAX technique can also be used to quantify elements detected so that the atomic and mass percentages of the elements can be extracted. This technique is non-destructive. It is a more compact and simple instrument compared to XMS and the electron guns required for illumination are readily available. Concentration distributions can be determined by scanning the electron probe over the surface of the sample. The spatial resolution is limited to about 5 μm by the electron beam,[24] which is an appropriate resolution for use with fiber preforms but is insufficient for determining RE distribution of fiber cores as diameters are typically of the order of 9 μm for single mode telecom fiber. Notwithstanding this, due to the obvious advantages that the EDAX offers, it has found use in a vast range of scientific fields including that of the production and characterization of optical materials.

EDAX is frequently used for the morphological and compositional analysis of a whole range of bulk optical products ranging from glasses or crystals to fiber preforms. In the latter case, there has been a noticeable volume of literature where it is stated that EDAX has been used for the determination of the dopants that compose the preform sample under examination. However, as previously stated, most often there is limited detail regarding the physical principles and operation of the EDAX technique and it is only the outcome of the analysis that is being presented.

A series of papers by Atkins *et al.*[24–26] examine the influence of co-dopants and the possible development of defects during the fabrication of optical fiber preforms with the help of the cathodoluminescence (CL) method. In that work, CL imaging was used in conjunction with EDAX and refractive index profiling to study fiber preforms fabricated using the MCVD process. The samples under examination were doped with a variety of typical elements such as Ge, P, Al, and Er, the concentration of which was determined in all cases by EDAX.

In a similar paper by Williams *et al.*[27] again using CL for the detection of defects in optical fiber preforms, it is stated that WDX had been applied for the elemental analysis of the samples. In other cases, EPMA is commonly used in conjunction with a scanning electron microscope for the determination of dopant distributions in fiber preforms.[28]

D. Fluorescence-induced laser scanning microscopy

Fluorescence-induced LSM is an excitation technique in which a laser source, tuned to one of the absorption bands of the RE dopants, is used to excite the sample under investigation. In LSM, the laser beam is focused onto the sample causing local excitation and the resulting fluorescence, which has a characteristic wavelength for each element, can then be detected. In contrast to the previously described systems, this technique is the simplest and the most cost effective since there is no need for bulky, complicated, and expensive equipment. In fact, it was due to the complexity and the high operation costs of the previously described techniques that feedback from dopant profiling was not actively pursued in the production of optical fiber. Consequently, researchers looked for alternative methods that are straightforward to implement and do not require complicated or specialist equipment.

A group of workers at the University of Southampton utilized this alternative technique for profiling erbium-doped fiber preforms in 1994.[21] In essence, their method involved the focusing of pump light into a spot of a few micrometers diameter incident on a doped region of preform. The amplitude of the fluorescence signal from this small region was then taken as an indication of the relative dopant concentration at that point, a process referred to as micro-fluorescence because of the small spatial dimensions involved. By scanning the pump spot across the diameter of a preform cross section, the fluorescence versus position data allowed the buildup of the relative erbium distribution profile.

Samples of Er doped fiber preforms (fabricated using the MCVD and solution doping techniques) about 5-10 mm in diameter and approximately 100 μm thick were examined by comparing the profiles obtained using fluorescence at wavelengths of 553 nm and 1500 nm. In each one of the detection schemes, both the lifetime and fluorescence signal profiles were acquired. The profile of the dopant distribution as a function of position across the core was obtained by taking a simple ratio of the fluorescence and lifetime. It was shown that

the dopant distribution is proportional to the signal profile $V(r)$ divided by the lifetime profile $\tau_m(r)$ according to the equation

$$N_0(r) = \frac{V(r)}{\tau_m(r)}(kAp\sigma_a)^{-1}, \quad (1)$$

where $N_0(r)$ is the total ion population, A is the Einstein coefficient, σ_a is the absorption cross section, and k is a constant. Here, r is the position characterized as the distance from the fiber central axis. Results revealed that the shape of the Er distribution was essentially independent of the core composition while the lifetime profiles varied. In the region where the ion environment was influenced by changes in the core composition, the lifetime exhibited a marked reduction from the values measured at the fiber core centre. It was also found that for Er concentrations in excess of about 0.1 wt. % (~350 ppm), there was a possibility that the 1500 nm fluorescence profile would be an inaccurate estimation of the true dopant distribution due to RE clustering effects. In that case, nearly resonant pumping at 514 nm with a resulting fluorescence wavelength of 553 nm was recommended for preforms doped at or above the aforementioned concentration. Overall, it was demonstrated that for measurements conducted under appropriate conditions, a simple but powerful profiling scheme could be used for profiling the erbium distribution in fiber preforms that involved little more than a laser diode, a translation stage, and a sensitive infrared detector.

The spatial resolution of this technique is limited by the excitation wavelength (improved spatial resolution for shorter wavelengths) and the numerical aperture of the focusing optics. However, this method of imaging produces a relatively large depth of field that could possibly cause interference by fluorescent dopants that are out of focus and away from the focal region. This reduces the contrast of the signal from the focal region and increases the spatial resolution to the order of a few microns, which results in the technique being most suitable for imaging bulk samples such as fiber preforms.

E. Evaluation of methods for optical fiber preform analysis

Summarizing the techniques described above in terms of their resolution, simplicity, and expense reveals that some require complex sample preparation techniques or complicated and high cost instrumentation, while others provide information that is not useful for most commercially available RE doped fiber since their operation is dependent on the RE dopant concentration being high. Nevertheless, from a simplicity and practicality point of view, the fluorescence induced LSM scheme clearly stands outs as the best candidate for the investigation of the dopant distribution of the Er ion in optical fiber preforms (Table I). On the other hand, chemical analytical based systems can provide information irrespective of the incorporated RE and its total dopant concentration. Furthermore, these systems can be also used for the analysis of other dopants (i.e., Al, P, Ge, etc.) normally present in the core of optical fiber preforms. The latter can be important in understanding the role of certain network modifiers and the impact they may have on the RE distribution profile or that of the refractive index of the fiber preform.

F. Limitations

The techniques described above may be applied to bulk samples, fiber preforms, or for large core diameter drawn fibers as is more common for high power fiber lasers.[29] Spatial resolution and practical limits prevent these techniques from being used to determine the concentration profile in conventionally drawn fibers. However, in analogy to refractive index measurements on optical fibers, it is common to use preform information and scale the data down to fiber dimensions after drawing. This assumes that the profile remains unchanged during the drawing process despite the fact that the preform is heated to high temperatures to permit drawing and the cross sectional area of the fiber is reduced in the process by more than three orders of magnitude. Huntington *et al.*[30] have shown that the assumption of scalability is not always valid for the determination of refractive index. Using scanning probe microscopic methods, it was demonstrated that there is a difference in the composition, and thus, the refractive index profile between preforms and drawn fibers. This is believed to be the consequence of various effects, such as the diffusion of particular elements that takes place during the drawing process. In essence, these effects alter the compositional profile of the drawn fiber in comparison to that of the preform. As a result, it is not always valid to simply relate the dopant concentration profile extracted from preform measurements to fiber dimensions.

TABLE I. Imaging techniques for the analysis of RE dopant distribution profiles in fiber preforms.

Techniques	Spatial resolution (μm)	Sample preparation	Instrumentation cost and complexity	RE specific	Other dopants	References
SIMS	10	Required	High	No	Yes	9, 17, and 22
XMS–EXAFS	100	Required	High	No	Yes	23
EPMA–EDAX–CL	5	Required	Average	No	Yes	19, 20, and 24–28
Fluorescence LSM	Wavelength and imaging optics dependent[a]	Not required	Low	Er only	No	21

[a]For a conventional wide-field microscope free of aberrations employing a 514 nm pump and a 100× MO (NA = 0.9), the ultimate lateral resolution is of the order of 0.5 μm. However, its axial resolution (depth of field) is noticeably inferior to that obtained employing a confocal arrangement.

III. RE DOPANT DETERMINATION IN OPTICAL FIBERS

It is evident therefore that potentially there are benefits in developing alternative analytical tools for the direct characterization of optical fibers. Techniques that have been developed in recent years specifically to achieve high resolution analysis of RE-doped optical fibers are summarized in this section. Similar to the case of fiber preforms, some of the techniques employed to reveal the location of REs in optical fibers are based on methods that are commonly applied in the field of analytical chemistry and material science. However, considerable recent effort has been invested in identifying and developing optical imaging systems that offer the potential to directly investigate the RE dopant distribution in optical fibers. These devices can be considered forerunners to laboratory based instruments that might one day be located in all optical fiber fabrication facilities as is now the case for the determination of the refractive index of a preform.

A. Transmission electron microscopy (TEM)

In TEM, a very high energy electron beam is focused on a sample that is thin enough to be partially transparent to electrons so that an image of the sample can be formed. The sample under examination by the TEM method can be magnified by a factor of more than 100 with a spatial resolution that can be better than 2 nm. Furthermore, the use of the electron beam can induce excitation of the atoms that constitute the sample. The resulting de-excitation of the atoms produces characteristic X-rays related to the elemental composition of the sample under investigation. In a similar way to EPMA based methods, the emitted X-rays can be detected and an elemental analysis of the sample can be obtained.

By utilizing this technique, Pugh *et al.* investigated the elemental diffusion during the drawing and heat treatment of a variety of optical fibers.[31] Samples of Ge doped, Er doped, and cadmium-sulpho-selenide-doped optical fibers were analyzed and the concentration profiles of the various dopants were obtained. The resultant combination of low beam spread in the sample (due to the sample thinness) and the high quality condenser lenses incorporated in the TEM arrangement produced line-scans showing the relative dopant distribution with a spatial resolution of the order of 85 nm. However, it was found that for the case of Er, only fibers doped at a concentration greater than ~1 wt. % (~3500 ppm) produced meaningful line-scans. In addition, it is critical that the sample under investigation is thin enough to be transparent to the electron beam. For this reason, a highly specialized technique is required to prepare thin (~400 nm) sections of optical fiber.[32] The standardized technique developed involves the use of a variety of other sample preparation methods such as mechanical polishing, resin-embedding, and ion-beam thinning. Nevertheless, due to the dimensions and the physical status of the glass optical fibers, a number of problems such as specimen damage or resin dissolution were encountered, which added to the complexity and time inefficiency of the sample preparation technique.

B. Micro-Raman and micro-fluorescence spectroscopies

A non-destructive method for measuring the relative dopant concentration of Ge-doped, P-doped, Er-doped, and praseodymium (Pr) doped silica based optical fibers was presented in the work of Benson *et al.*[33] Their method involved a combination of Raman and fluorescence spectroscopy techniques so that either the Raman spectra of Ge and P or the backscattered fluorescence of Er and Pr could be detected using the same system. Detection of data was accomplished with a Dilor XY multichannel spectrometer with a microscope attachment, while a 488 nm laser source was used to excite the samples. For the case of Ge doped and P doped fibers, relatively thin fiber slices (~50 μm thick) were first prepared for analysis, while the RE doped fibers were examined from the side.

The laser was focused at the core-cladding interface and the fiber stepped horizontally in 1 μm steps, such that the laser scanned the diameter of the core. The backscattered Raman or fluorescence spectra were recorded for each fiber position so that a one-dimensional relative distribution profile could be built up for each one of the dopants. It was assumed that the Raman and fluorescence intensity was proportional to the local dopant distribution. For RE-doped fibers at high doping concentrations, this assumption may be invalid as concentration dependent cooperative processes can alter the relaxation process (hence, producing a change in the fluorescence intensity). The authors also assumed that the focal position of the laser did not vary with fiber position, which may not be the case since fluorescence intensity detection systems are sensitive to alignment that can be affected by mechanical vibrations and beam drift.

Using a similar experimental arrangement to the one just described, and by developing an alternative way of relating Raman and fluorescence signals to RE dopant concentration, the characterization of neodymium (Nd) doped fluoride glasses was achieved by Fornoni *et al.* The method was further used to deduce concentration measurements of Er doped optical fibers.[34] In both projects, the RE dopant concentration was obtained by comparing the intensity of a fluorescence line emitted by the RE ion with the intensity of the Raman line from the host matrix. The ratio of the fluorescence signal to the Raman signal is proportional to the dopant concentration. In the latter work, studies of both preforms and drawn fibers that had been doped with Er at 1000, 2000, and 5000 ppm were performed and compared. The experimental configuration employed for the detection of the signals involved a microscope (Olympus BHT) connected to a spectrometer (Dilor Z24 single channel, triple monochromator) that was also equipped with a photomultiplier. The 476.5 nm argon line was used for the excitation of the samples since it was the only one that could induce a strong Raman peak that did not interfere with backscattered luminescence lines.

Among the many luminescence lines that have been observed in Er doped ZBLAN glasses, only a few exhibit a linear relationship with the excitation power. As a result, it is of great importance that the right excitation wavelength is selected and that the appropriate fluorescence spectra are

detected. In addition, a calibration curve, determined from samples with known concentration, is required in order to deduce the concentration of an unknown sample from the measurement of the ratio fluorescence signal to the Raman signal. Consequently, this technique is not self-calibrated, additionally the final result will not be reliable if the depth of field of the focus is not strictly controlled.

C. Fluorescence intensity confocal optical microscopy (FICOM)

An analogous technique to the laser scanning and micro-fluorescence microscopies outlined in Sec. III B was employed in conjunction with a confocal microscope in order to map the relative concentration profile of Er doped fibers.[35] FICOM assumes that the measured fluorescence intensity detected by the confocal microscope is proportional to the local dopant distribution. One year later, this technique was expanded by introducing a phase sensitive detection scheme to improve sensitivity and obtain a better signal-to-noise ratio.[36] In both schemes, the 488 nm argon line was used for the stimulation of the samples while the intensity of the 565 nm fluorescence line was considered as an indication of the local erbium dopant concentration. The confocal microscope (model Biorad MRS 600) used for the detection of the backscattered fluorescence obtained high resolution linescans of the endface of a cleaved fiber by working in the fluorescence detection mode. The 40× objective lens (0.55 NA) that was employed in the experimental configuration of the early version of this technique resulted in a 0.33 μm spatial resolution, while in the latter case, a 100× (unknown NA) objective produced a spatial resolution of 0.25 μm.

In both experiments, the intensity profile was obtained by scanning the excitation beam across the centre of the fiber core. The acquired intensity profiles were then related directly to the relative erbium ion distributions without establishing that the resulting backscattered fluorescence signal is indeed proportional to the Er ion concentration. Care must be taken regarding the reliability of the intensity based measurements given that cooperative effects in RE doped fibers may yield incorrect values of dopant concentration.[21]

A similar imaging concept employing a commercial Renishaw Raman microscope was also used to investigate the Er ion distribution in a range of optical fiber samples (Fig. 1).[37] In contrast to the work outlined in Refs. 35 and 36 where the 488 nm line was used as the excitation source, in this latter case, information about the erbium ions in the investigated samples was achieved through a near resonant exciting scheme using a 514 nm line. The use of a 514 nm laser line as opposed to the 488 nm line ensured that more ions can be promoted to the upper energy levels and therefore a higher signal to noise ratio was achieved. Consequently, shorter integration times allowed the acquisition of high spatial resolution (around 0.4 μm, using a 100× –0.9 NA microscope objective) transverse linescans as well as two-dimensional images of the relative Er ion distribution in the fibers under analysis. In addition, by exploiting the high wavelength resolution of a Raman spectrometer, adequate wavelength discrimination between the pump and backscattered fluorescence signals was obtained, while the reduced

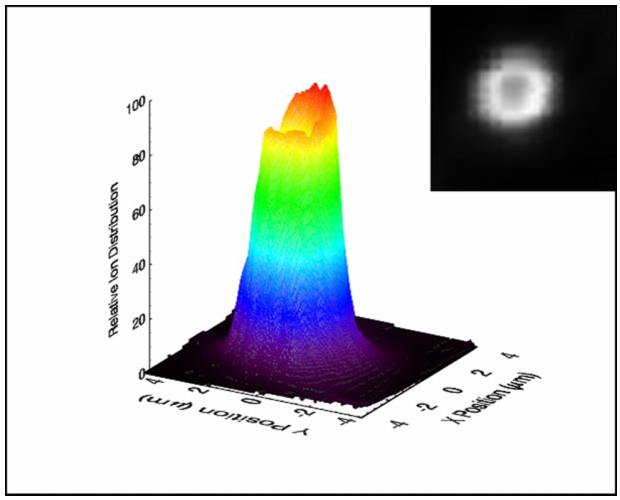

FIG. 1. A two dimensional erbium concentration map across the core of an optical fiber reproduced with permission from Ref. 37. The profile clearly shows the central dip in the erbium concentration caused in the preform collapse process. Reprinted from F. Sidiroglou *et al.*, Opt. Express **13**(14), 5506-5512 (2005). Copyright 2005 Optical Society of America.

depth of field of the confocal microscope ensured that measurements were free from possible guided fluorescence effects from within the fiber. This technique requires no sample preparation other than the standard cleaving of the fiber end.

More importantly, the reliability issue of the intensity based measurements (given that cooperative effects in RE doped fibers may yield incorrect values of dopant concentration) was also addressed.[38,39] A simple theoretical model consisting of an ensemble of rate equation systems, characteristic of the most likely electronic transitions that take place in the vicinity of Er doped silica glasses, was developed and solved. Through this theoretical study, it was established that information about the relative Er ion distribution in fibers could be inferred by simply monitoring the backscattered fluorescence signal originating from the de-excitation of the specific energy levels used in the investigated samples.[37] Furthermore, the validity of the proposed theoretical model was confirmed through a comparison of the Er ion distribution profiles acquired using the FICOM technique and those obtained from the application of a powerful analytical ion probe.[37–39,44]

D. Fluorescence lifetime confocal optical microscopy

Among the fluorescence intensity based techniques used for the determination of RE dopants in optical fibers and preforms, it has been emphasized that a possible ambiguity in the measurements may arise when RE ion cooperative effects take place within the vicinity of the host matrix. A technique was developed where lifetime instead of intensity measurements has been used to determine concentration profiles in optical fibers.[40] This method was based on measurements of relaxation rates of the $^3P_0$ excited state in Pr doped fibers.[41] The extent that cross-relaxation processes altered the relaxation rate of the $^3P_0$ level was investigated and was shown to be an accurate indication of absolute dopant concentration. The experimental arrangement included a confocal microscope for the acquisition of the intensity and the time resolved fluorescence line

observed at a wavelength of 635 nm. An argon ion laser at 476.5 nm was used to excite the samples, while the decay fluorescence curves were recorded on an oscilloscope. Lifetime information was gathered across the entire fiber endface so that a two-dimensional image of the dopant concentration could be built up for the whole fiber core section. This scheme has the advantage compared with the previously described fluorescence intensity confocal optical microscopy in that it is not dependent on the intensity of the detected fluorescence. As a result, this technique is less sensitive to the optical properties of the imaging system. In addition, it does not require reference intensity levels from fibers of known concentrations since the measured lifetime can be related directly to absolute dopant concentration. The point by point acquisition of lifetime data does increase the time required to conduct a measurement in comparison to intensity based techniques.

E. Near-field scanning optical microscopy (NSOM)

NSOM is known to be capable of attaining sub-wavelength resolution and as a result it is an excellent candidate for the characterization of objects with small dimensions such as the core of an optical fiber. Trautman *et al.*[42] initially investigated the potential of NSOM for fluorescence contrast imaging of dye samples.

In this work, 488 nm pump light was launched into a near field probe that was positioned close to the surface of a microscope coverslip, coated with a dye solution, which enabled spatially resolved images of the fluorescence. The NSOM fluorescence image contrast technique was then applied to a 500-nm-thick section of an Er doped optical fiber. The 1.5 μm induced fluorescence was detected so that an image of the relative Er ion distribution could be obtained. However, due to the small thickness of the sample and the relatively low erbium concentration, a larger aperture NSOM probe was required for the detection of the weak signal. Consequently, the spatial resolution of the resulting image was in the submicron range (~0.2 μm), similar to that obtained using FICOM scheme. Furthermore, the backscattered fluorescence signal at 1.5 μm resulting from the de-excitation of the $^4I_{13/2}$ energy level cannot be related directly to the Er concentration since the dynamics of the $^4I_{13/2}$ state are strongly affected by ion to ion interactions and in particular by ESA (Excited State Absorption).[38]

As a result, NSOM studies of Er doped optical fibers were attempted by monitoring the backscattered fluorescence signal originating from the de-excitation of the $^4S_{3/2}$ energy level,[38] which has been found to be independent of possible inter-ionic effects.[37–39] It was recently also shown[43] that initial concerns regarding practical limitations such as weak fluorescence signal and stray light contaminations can be overcome and that good qualitative agreement can be established on data acquired from different techniques (Fig. 2).

F. Time of flight secondary ion mass spectroscopy (ToF-SIMS)

ToF-SIMS is another spectroscopic imaging technique that has been used for the determination of dopant concentration profiles in optical fibers and that has borrowed once more from the field of analytical chemistry and material science. It is an extension of the SIMS system described previously. In a similar manner to the conventional SIMS method, ToF-SIMS is a surface analytical technique that uses an ion beam to remove small numbers of atoms from the outermost atomic layer of a surface under analysis. A short pulse of primary ions strikes the surface, and the secondary ions produced in the sputtering process are extracted from the sample surface and into a time-of-flight mass spectrometer. This technique is capable of generating an image with spatial resolution in the order of 0.5–1 μm, which is a considerable improvement to the 1-2 μm spatial resolution of the SIMS system. ToF-SIMS was employed for the investigation of dopant diffusion in Ge doped, P doped, fluorine (F) doped, and RE doped optical fibers.[44] Freshly cut fiber sections were analyzed using a Physical Electronics TRIFT 2100 ToF-SIMS instrument with an isotopically enriched $^{69}Ga^+$ liquid metal ion source. In the case of RE doped optical fibers, a set of experiments was performed and relative compositional profiles of Er-ytterbium (Yb) co-doped ions were obtained with a spatial resolution of approximately 0.5 μm. From the acquired images, additional information about the dimensions of the fiber core and the erbium/ytterbium concentrations ratio was also deduced.

In a more recent publication, it was shown that a compact orthogonal ToF SIMS detector attached to a FIB-SEM system can be used to analyze cross sections of optical fibers. In addition, an attempt was also made to quantify the optical fiber SIMS profiling by simply calibrating the SIMS profiles against the EDX acquired data.[45]

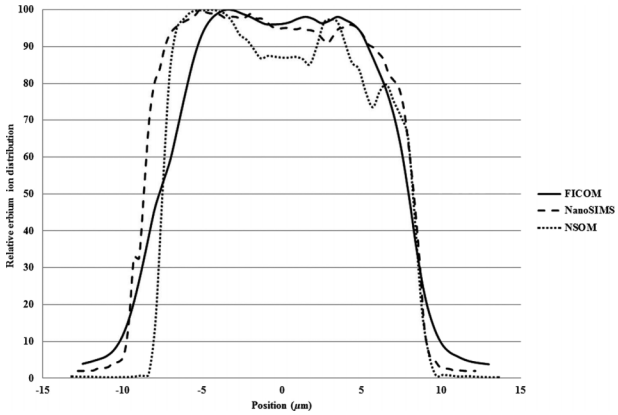

FIG. 2. Normalised transverse profiles through the center of the fiber showing the relative erbium ion distribution in the same EDFA sample, acquired from the application of the microfluorescence (continuous line), nano secondary ion-mass spectrometry (NanoSIMS) (dashed line), and NSOM (dotted line) techniques. Reprinted from F. Sidiroglou, A. Roberts, and G. W. Baxter, Opt. Eng. **53**(12), 126104 (2014). Copyright 2014 Society of Photo Optical Instrumentation Engineers (SPIE).

G. Nano secondary ion mass spectroscopy

RE dopant distribution profiles have been also obtained with the application of another SIMS based analytical scheme, known as Nano Secondary Ion Mass Spectroscopy or Nano-SIMS.[46] In contrast to ToF-SIMS, which operates under static data acquisition conditions, Nano-SIMS implements a dynamic acquisition approach where higher sputtering

rates are used and a depth characterization of the sample is accomplished. ToF-SIMS uses an electric field to accelerate the ions through the same potential, and then, measures the time the ions take to reach the detector. If the particles all have the same charge, then their kinetic energies will be alike, and their velocities will depend only on their masses. Consequently, by monitoring the time it takes for each particle to reach the detector, information about the composition of the investigated surface can be collected.

Dynamic SIMS on the other hand employs either a magnetic sector or a radio-frequency quadruple mass analyzer that monitors the different trajectories that each ion follows due to an applied electric or magnetic field. The ions enter a magnetic or electric field, which bends the ion path in a unique trajectory for each ion species that is characteristic of their mass-to-charge ratios. The ions eventually reach the detector and their relative abundances are measured.

At the sample region and in contrast to conventional SIMS, where individual objective and extraction ion optics are employed for the focusing of the primary beam and collection of the secondary sputter, respectively, with the NanoSIMS a co-linear optical scheme accomplishes both tasks. As a result, the dual-functioning optics can be brought to close proximity to the sample resulting in superior spatial resolution (a few tens of nm) and high collection efficiency. In addition, the commercially available Nano-SIMS systems (like the Cameca NanoSIMS-50 ion microprobe used in the work presented in Ref. 46) incorporate an array of five (some seven) discrete-dynode-type (Hamamatsu, Cu-Be) electron multiplier detectors that can provide the simultaneous acquisition of up to five different ion species (Figs. 3 and 4).

Using this ion microprobe, a full simultaneous multi-dopant investigation of the fiber core and the adjacent cladding region was achieved.[46] The relative distribution profiles of the RE (Er or Yb), Ge, Al, P, and background Si in the fiber core were simultaneously acquired with submicrometre resolution. In addition to transverse line scans through the center of the fiber samples, two-dimensional images showing the relative distribution of all the relevant elements present in the fiber core area were also presented. The fiber samples were first mounted in specially designed holders so that the end face of the fibers could be polished and before they were gold coated to provide surface conductivity. Measurements were performed by using chemically reactive primary ions of $^{16}O^-$ with impact energies of 16 keV, while the resulting sputtered secondary ion beam was mass analyzed and recorded by an array of five electron-multiplier detectors. Each detector was used to record one of the following species: $^{27}Al^+$, $^{70}Ge^+$, $^{31}P^+$,

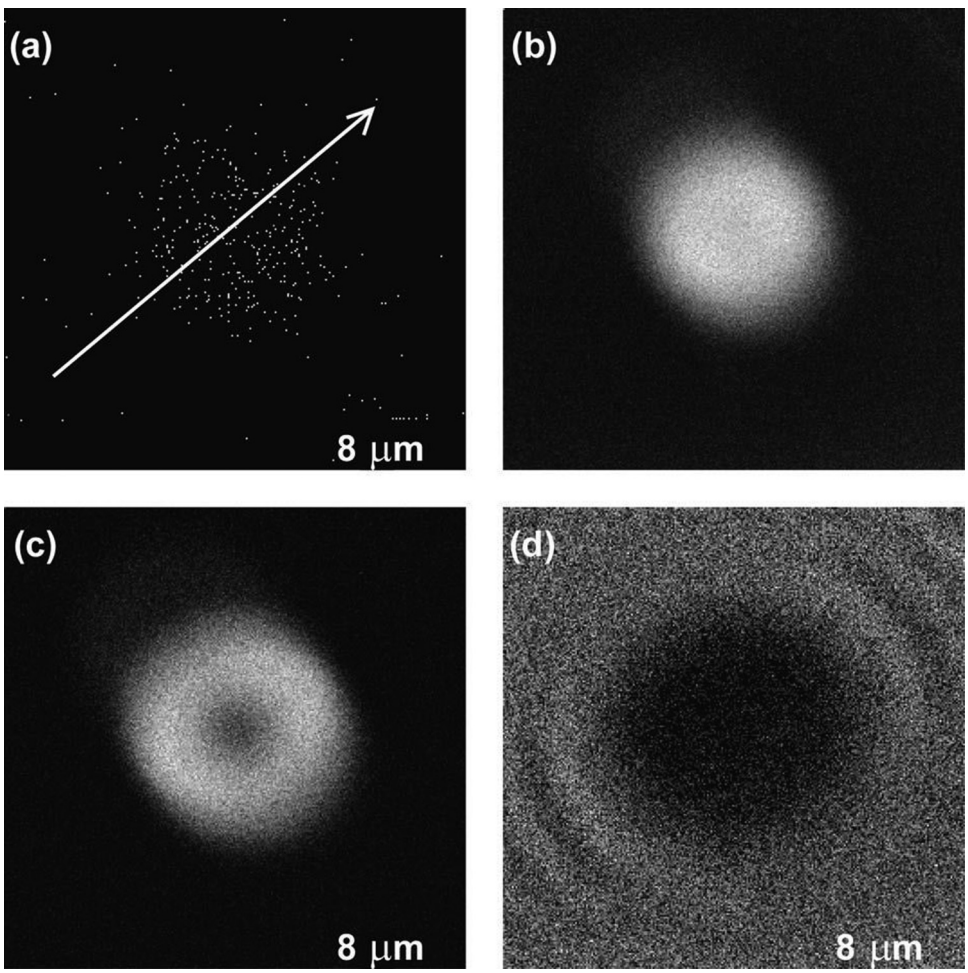

FIG. 3. Cross section of the relative ion distribution in the core of an EDFA sample (a) Er, (b) Al, (c) Ge, and (d) P. The arrow indicates the direction along which subsequent line scans were obtained. Reprinted with permission from F. Sidiroglou *et al.*, Opt. Lett. **31**(22), 3258-3260 (2006). Copyright 2006 Optical Society of America.

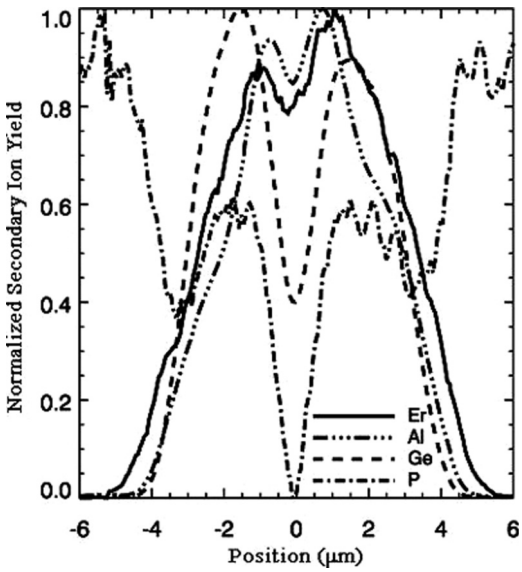

FIG. 4. Transverse line scan through the center of an Er doped optical fiber sample showing the relative ion distribution of all dopants present in the fiber core. The endface of the fiber was scanned by a 0.4 μm ion probe using an integration time of 10 s per step. Reprinted with permission from F. Sidiroglou *et al.*, Opt. Lett. **31**(22), 3258-3260 (2006). Copyright 2006 Optical Society of America.

$^{44}[SiO]^+$, and either $^{166}Er^+$ in the case of erbium doped fibers or $^{174}Yb^+$ in the case of ytterbium doped fibers. As a result, simultaneous acquisition of all dopants present in the central fiber core area was achieved (Fig. 4). Valuable information regarding the solubility and diffusion characteristics of various elements typically present in the core of RE-doped optical fibers was gathered.

Employing similar NanoSIMS instrumentation (NanoSIMS 50L), the composition of nanoparticles diluted in a silica glass matrix in the core of optical fibers was successfully assessed with a demonstrated resolution between 60 and 120 nm.[47]

H. Atom probe tomography

The most recent advances in the measurement of RE doped optical fiber have been motivated by the desire to gain a better understanding of the intentional formation of amorphous dielectric nanoparticles within the fiber core.[48] Atomic Probe Tomography (APT) has been demonstrated to measure compositional changes for very small particle sizes (~1-10 nm) where it was shown that the erbium ion had a preference to be located within the nanoparticles (rich in Mg and P). The images display a very high spatial resolution enabling the identification of nanoparticles of dimensions less than 10 nm; however, the concentration of erbium ions is around 0.05 at. % making their identification difficult other than correlating, on average, their position relative to a nanoparticle.

IV. DISCUSSION

Like in the case of studying the RE dopant distribution in fiber preforms, the techniques employed to reveal the location of REs in optical fibers can be divided in two main categories: systems that are commonly applied in the field of analytical chemistry and material science (TEM, Micro-Raman, the SIMS counterparts, and APT) and those that employ optical based schemes (FICOM, FLCOM, and NSOM).

From a spatial resolution point of view, it can be observed that submicron information can be obtained from the application of most of the reviewed imaging schemes (Table II), with APT offering the highest possible resolution of better than 10 nm. However, such instruments are rare and costly thereby being well beyond the reach of most laboratories. TEM is more readily available and depending on the experimental configuration and the concentration of the RE ion can achieve a resolution better than 100 nm. In addition to this, both the APT and TEM can be used for the analysis of any dopant present in the core of the investigated fiber samples, which is critical in understanding the relationship between the REs and other network modifiers. The limit of such instruments in providing information for specific REs is difficult to quantify with, for example, the ATP effectively counting individual ions within a defined volume with sub-nanometer boundaries. Determining the resolution then becomes a statistical issue requiring a certain number of ions within a volume to obtain a particular confidence limit. Both ATP and TEM require the application of a complicated sample preparation stage and the use of high cost and complex instrumentation. The latter is characteristic of all chemical analytical based methods.

On the other hand, the main advantage of the optical microscopic imaging schemes lies in their simplicity and ease of use. In contrast to the chemical analytical methods, which require bulky, relatively expensive, and complex components as well as time consuming and complex operation, an optical approach represents a much cheaper and more practical

TABLE II. Imaging techniques for the analysis of RE dopant distribution profiles in optical fibers.

Techniques	Spatial resolution (μm)	Sample preparation	Instrumentation cost and complexity	RE specific	Other dopants	References
TEM	0.085	Required	High	No	Yes	30 and 31
Micro-Raman	1	Required	Average	Yes	Yes	32 and 33
FICOM	0.33	Not required	Low	Er only	No	34–38 and 42
FLCOM	~1	Not required	Low	Pr only	No	39 and 40
NSOM	0.2	Not required	Low	Yes	No	37, 41, and 42
ToF-SIMS	0.5-1	Required	High	No	Yes	43 and 44
Nano-SIMS	0.2-0.4	Required	High	No	Yes	37, 45, and 46
APT	0.010	Required	Very high	No	Yes	46

alternative. High quality results can be achieved in principle using components (i.e., argon ion laser sources, confocal optics, interference filters, light detectors, etc.) that are commonly available in most optical laboratories. In addition, non-destructive sample measurements can be obtained directly from the freshly cleaved end-face of optical fibers without the need for special sample preparation techniques.

The main drawback of these optical based methods is that their operation is limited to the investigation of specific RE elements. The focus of this review has been on the important erbium ion; however, other REs may be examined in a similar manner provided that certain spectroscopic characteristics can be linked with the distribution of the investigated RE ions and as long as any fluorescence based measurements are not limited by diffraction in terms of spatial resolution. Assuming that such relationships can be established theoretically for each particular RE, the use of a NSOM imaging scheme would overcome this practical limit. However, further work is required in making the NSOM technique more robust in terms of signal to noise ratio and its susceptibility to stray light contamination. Likewise other super resolution techniques, as has been applied with the field of bio-imaging, may represent realistic options for improving the measurement resolution and grant access to spectroscopic information on individual nanoparticles. Caution is required for all optical methods during data acquisition since any sample misalignments, laser power variations, or transition stage malfunctions could lead to ambiguous outcomes, especially in the case of the FICOM scheme.

The operation of the SIMS based arrangements can be susceptible to possible ion beam misalignments or other effects that may have a negative impact on data acquisition. These methods, however, can in theory provide information about any ion species from the periodic table of elements and it can be therefore employed for the investigation of any element normally present in the core of optical fibers. It has to be emphasized, however, that different elements respond in a different way to the primary ion beam and therefore the induced secondary ion signal can vary substantially between different ion species. The final spatial resolution is also dependent on the type of primary ion source used in the investigation, which is determined by the ionic nature of the elements to be studied.

The main advantage of the NanoSIMS system, however, which is a direct consequence of its design and instrumentation, is the fact that it allows the simultaneous detection of up to five different ion species. The latter ensures that a full elemental characterization of the core of optical fibers can be obtained. As a result, valuable information regarding the RE distribution plus that of other elements can be extracted, while the relationship between all elements can be further evaluated. It is possible that, given elemental standards can be used in order to calibrate the system, absolute concentration profiles for each element can be obtained.

V. CONCLUSION

This review has described the various techniques used to determine the erbium dopant distribution of optical fiber. The two main categories of techniques investigated are those that characterize the fiber preform and those that measure directly the fiber core. It has been argued that on the basis of economy, simplicity, and practicality, the fluorescence based schemes constitute the superior techniques, while the NanoSIMS represents the better alternative when a thorough sample examination is preferred. It is suggested that the use of a NSOM system could be extremely beneficial for the investigation of any RE as long as its operation can be optimized in terms of signal acquisition.

Recent innovations in RE doped optical fiber fabrication are looking to exploit the potential of RE rich nanoparticles within the core of the fibers. The dimensions of the nanoparticles are believed to be as small as 1 nm and as large as 500 nm. Researchers have used NanoSIMS and ATP to characterize the elemental distribution in such fibers but the search is on to better understand the fluorescence and lifetime characteristics of individual nanoparticles. The optical techniques described here have insufficient resolution to resolve all but the largest nanoparticles and hence further development is required to meet this challenge. It is foreshadowed that super-resolution imaging techniques as have been developing in the bio-imaging field are a likely successor to the NSOM imaging systems as the best systems for resolving RE distributions in drawn optical fiber.

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